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(54) MOULD PREPARATION

(71) We, IMPERIAL CHEMICAL INDUSTRIES LIMITED, Imperial Chemical House, Millbank, London SW1P 3JF, a British Company, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to compositions for use in mould casting and to methods employing such compositions.

In investment casting, a shell is built up around an expendable pattern, which is usually of wax, by dipping the pattern into a slurry of a binder and a refractory material and drying the coating. This process is repeated a number of times until a covering of a desired thickness and strength is formed. Generally the dipping process has to be repeated four, five and possibly up to eight times, usually with a drying or curing step between each dipping and application of intermediate coats of refractory powder.

Our copending British Patent Application No. 48575/71 Serial No. 1,408,266, describes an improved method of investment casting enabling the use of a reduced number of processing steps, the process involving, essentially, the use of a thixotropic dipping mixture comprising a refractory material, a binder, a liquid dispersant for the binder and a thickening agent which renders the composition thixotropic.

The present invention is applicable to mould preparation by the shell process of investment casting in which an expendable pattern is dipped into an investment coating composition, removed from it, the adherent coating dried and the pattern removed.

It has sometimes been found that with existing processes there is a tendency for the mould to crack due to drying contraction. At

the least the strength of the mould may be affected adversely by such cracking.

We have found that cracking of the mould may be reduced, or even obviated completely by incorporating fibres in at least one of the coating layers.

The invention, therefore, provides a method of preparing a mould which comprises the steps of immersing a disposable pattern into a composition comprising a particulate refractory material, a binder, a liquid dispersant and fibres, removing the coated pattern from the composition, drying the adherent coating and removing the pattern.

Preferably the composition has thixotropic properties and according to a preferred embodiment the invention provides a method of preparing a mould which comprises the steps of immersing a disposable pattern into a composition comprising a refractory material, a binder, a liquid dispersant, fibres and a thickening agent which renders the composition thixotropic, removing the coated pattern from the composition, drying the adherent coating and removing the pattern.

The fibres employed according to the invention are such as will not be affected undesirably by the conditions employed either during formation or use of the moulded product. In particular they may be resistant to the extremes of heat to which the mould is subjected in use, although since their reinforcing action is particularly important during the drying and early use of the mould, their gradual deterioration or destruction subsequent to complete hardening of the mould may be tolerated and may even be desirable as will be explained subsequently.

However, the fibres employed will preferably be completely resistant to deterioration under the conditions of use, and as examples of such resistant fibres we may mention those

based on the oxides, nitrides and/or carbides of Be, Mg, Ca, Ti, Zr, B Al and Si; carbon fibres, metal whiskers and chopped metal wires may also be suitable. Glass fibres may also be employed in the compositions used in the present invention.

It is important to select fibres of appropriate dimensions, since if they are excessively long the compositions may not run freely and evenly and they may not follow the contours of the pattern satisfactorily. Thus we have found for a smooth casting the maximum length for the fibres is of the order of 6 mm but for a casting having sharp corner or holes or any intricate detail the fibre length should not exceed about 3 mm. Preferably the fibres are 1—2 mm in length. The diameter of the fibres may be from 0.01 to 0.5 mm and preferably is from 0.1 to 0.3 mm.

The proportion of fibres in the composition generally should not exceed 10% by weight of the solids content of the composition; preferably they form 0.1 to 4% and more preferably 0.5 to 2% by weight.

Mixing of the fibres into the composition is preferably carried out using relatively gentle mixing means to avoid undue breakage of the fibres, although the possibility of incorporating long fibres (that is, fibres longer than will be required in the final composition) and then chopping them to the desired length in the composition is not excluded.

It is sometimes desirable for a mould to have a degree of porosity, and to attain this we have found it convenient to include in the composition fibres which subsequently can be degraded, for example by incineration, to give such porosity. Thus we have included in investment compositions fibres of polyethylene terephthalate and of Nylon, which fibres were subsequently destroyed by heating the formed mould to a suitably high temperature. Such degradable fibres do, of course, impart strength to the mould while they retain their integrity, and may therefore serve a dual function first of reinforcing the mould and on destruction of introducing a degree of porosity. It will be appreciated that heat degradable fibres may be used in addition or in place of thermally resistant fibrous material.

The fibres chosen for use according to any aspect of the invention will not of course be such that their presence is on balance disadvantageous. The fibre surface may be treated to improve its surface properties in known fashion, for example to impart or improve the bond strength with the matrix of the mould.

In order to maintain even distribution of the fibres during dipping of the pattern into the composition it is desirable that the composition is agitated, and this is particularly necessary when a thixotropic composition is employed; in the latter case, the thixotropic composition should in particular be subjected

to shear conditions, at least in the region of the pattern. Agitation may be effected by any convenient means, for example, by stirring the mixture, by vibrating the pattern and/or the composition or by vibrating the vessel holding the composition.

Particularly when thixotropic compositions are used the shear conditions reduce the viscosity of the mixture thereby causing it to flow easily around the pattern. The shear conditions are therefore particularly desirable when the pattern is of a complex shape and/or the mixture is highly viscous.

Preferably, the thixotropic composition is such that a shear stress of at least 1000, preferably 3000 and more preferably 5000 dynes/cm² (as measured on a Contraves Rheomat 15 rheometer ("Contraves" is a Registered Trade Mark) fitted with "D" cup and bob) is required to break the structure of the set-up composition from a solid to a liquid.

It is generally desirable to give the pattern a primary, relatively thin coating in a refractory mixture containing no fibres or thickening agent.

Suitable particulate refractory materials include silica; alumina, e.g., tabular alumina and bauxite; magnesium, calcium and titanium oxides zinc and tin oxides; magnesite; mag-chrome grog, zirconium silicate; zirconia; aluminium silicates, e.g. sillimanite, andalusite, kyanite, mullite and Molochite (Registered Trade Mark); porcelain and china clays; carbides e.g. silicon and tungsten carbide; nitrides, e.g. silicon and boron nitride; boron; ferric oxide; chromium oxide; chromite; mica.

Generally the refractory material is in the form of a powder. It is preferred to use a refractory powder at least 50% by weight of which has a particle diameter of less than about 0.15 mm and more preferably less than 0.075 mm.

A wide variety of binders may be used including any conventionally used for investment casting. In many cases it may be desirable to use a mixture of two or more binders. Examples of suitable binders include silicates, e.g., alkyl silicates, such as ethyl or isopropyl silicate, aminoalkyl silicates, monoethanolamine orthosilicate, alkali metal silicates such as sodium and/or potassium silicate, silica sols, metal oxychlorides such as aluminium oxychloride, gypsum/silica mixes and cements such as aluminous or Portland cement. Silica sol binders are preferred. Metal phosphates, e.g. of aluminium, are other binders, and they may be simple or complex phosphates. Examples of suitable simple phosphates include aluminium orthophosphates e.g., $\text{Al}(\text{H}_2\text{PO}_4)_3$; $\text{Al}_2(\text{HPO}_4)_4$ and AlPO_4 . $\text{Al}(\text{H}_2\text{PO}_4)_3$ is particularly suitable. AlPO_4 may be conveniently used in a mineral acid solution, e.g., hydrochloric or nitric acid.

Of the complex aluminium phosphates those described in our U.K. Patent Specification No. 1,322,722 may be mentioned.

Since the composition is employed in liquid form it comprises a liquid dispersant for the solid components.

The liquid dispersant is preferably also a solvent for the binder. Choice of a dispersant having solvent properties clearly depends upon the type of binder to be used, but generally suitable solvents include polar solvents, for example, water; mineral acids; alcohols, e.g., aliphatic alcohols, especially methanol, ethanol, isopropanol and butanol; glycols and glycol ethers, e.g., ethylene glycol monoethyl ether. Mixtures of two or more solvents may be used.

Where used the thickening agent can be any which will give a thixotropic mixture of the desired viscosity. Suitable agents include

clays, e.g., the naturally occurring magnesium silicate types such as bentonite and fuller's earth. Particularly preferred are the treated clays, for example highly beneficiated hydrous magnesium silicate e.g., that sold under the trade mark "Ben-a-gel", and the organically modified magnesium silicate clays, e.g., those sold under the trade mark "Bentone" of which the grades LT; 18C; 34; 38 and 27 are particularly suitable. Other thickening agents include clay substitutes, such as cellulose ethers, e.g., methyl cellulose, ethyl methyl cellulose, hydroxypropyl methyl cellulose, fumed silica, e.g., that sold under the trade mark "Cab-o-sil", and some starches.

The relative proportions of the components of the investment compositions can vary over a wide range. Suitable percentages by weight of the mixture for the components may be as follows:—

	Refractory material	10—90% preferably 50—80%
	Binder (dry solids content)	1—15% preferably 5—15%
	Dispersant	5—50% preferably 10—30%
	Fibres	0.01—10% preferably 2—8%
45	Thickening agent (where present)	0.05—10% preferably 0.2—5%

The green mould may be dried before firing in a stream of air or by heating at a moderate temperature, for example up to 50°C. It is usually desirable to dry slowly, e.g., for 4 hours to 3 days, at temperatures of 10 to 30°C. If an expendable pattern is used and it is of sufficiently low melting point, it may be drained in its molten state from the mould. This may be achieved by shock heating the mould, e.g., to a temperature of at least 750°C, preferably 900 to 1100°C. Such heating may be continued to cure the mould. Alternatively the mould may be treated with steam, preferably dry steam, at above atmospheric pressure, e.g., from 2 to 10 atmospheres. An expendable pattern may also be removed by dissolving it in a suitable solvent, for example trichloroethylene or perchloroethylene.

However, generally the moulds are cured by heating at temperatures of at least 80°C, for example in the range of 150 to 1500°C, pre-

ferably 800 to 1200°C, for a time sufficient to harden them e.g., from 5 to 60 minutes. An expendable pattern, or remnants thereof, may be substantially completely removed during firing.

In many cases it is desirable to treat the mould before curing with a hydrophobic substance, for example an alkyl silicate in a suitable solvent, e.g., an alcoholic solution of ethyl silicate, or a silane in a suitable solvent, e.g., an alkyl alkoxysilane, such as methyl triethoxysilane, in an alcohol, e.g., isopropanol. Alternatively or additionally the mould may be treated with a gas which reacts with the binder to form a gel, for example ammonia. This is particularly desirable when the mould is to be exposed to steam, before curing. It is also possible to treat with magnesium oxide to harden the surface.

The invention is illustrated in the following examples in which percentages are by weight.

Example

Three compositions were prepared as follows:—

	A	B	C
Molochite 120 B.S. mesh	1020 gm	4200 gm	200 gm
Silica sol (30% SiO ₂ by wt in liquid dispersant)	720 ml	1800 ml	125 ml
95 "Benaqua"	20 gm	21 gm	1.5 gm
Water	170 ml		
Chopped Kaowool fibres ($\frac{1}{8}$ " long)	50 gm		5 gm
Zirconia fibre ($\frac{1}{8}$ " long)		47.5 gm	

"Benaqua" and "Kaowool" are Registered Trade Marks.

All three compositions were stirred vigorously to homogeneity and allowed to "set up" to give thixotropic products.

5 A wax pattern was first coated, by immersion, with a primer layer consisting of Zircosil (Registered Trade Mark) 200 mesh and silica sol; a stucco coat of Molochite 50/80 B.S. mesh was then applied and the primer coat allowed to dry. The coated pattern was then dipped in one of the above compositions 10 which during immersion was vibrated at 200 cycles/second by applying an oscillator to the base of the container.

15 The pattern was withdrawn coated with a layer of thickness about 0.5 cm which was allowed to dry at 30°C for 4 hours. The wax pattern was then removed by flash dewaxing at 1000°C for about 30 seconds. The resulting mould was then fired at 800°C for 20 20 minutes.

WHAT WE CLAIM IS:—

1. A method of preparing a mould which comprises the steps of immersing a disposable pattern into a composition comprising a particulate refractory material, a binder, a liquid 25

dispersant and fibres, removing the coated pattern from the composition, drying the adherent coating and removing the pattern.

2. A method according to claim 1 in which the composition comprises a thickening agent 30 which renders the composition thixotropic.

3. A method according to claim 1 or 2 in which at least some of the fibres in the composition are inorganic and resistant to incineration. 35

4. A method according to claim 1 or 2 in which at least some of the fibres in the composition are of heat degradable material.

5. A method according to any one of the preceding claims in which the fibres form up to 10% by weight of the solids content of the composition. 40

6. A method according to any one of the preceding claims in which the fibres have a maximum fibre length of 6 mm. 45

7. A mould prepared according to the process of any one of the preceding claims.

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